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IS 13840 (Part 1) : 1993

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भाग 1 प्रत्यक्ष दहन गुरुत्वमिति पद्धति द्वारा कार्बन ज्ञात करना

Indian Standard

CHEMICAL ANALYSIS OF FERROTITANIUM

PART 1 DETERMINATION OF CARBON BY THE DIRECT COMBUSTION GRAVIMETRIC METHOD

UDC 669·15·782-198·543·21 [546·26]

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Part 1) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferro alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorous, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard the Sectional Committee decided that a separate series for each ferro alloy be prepared. Accordingly, IS 1559 in its various parts was published for the methods of chemical analysis of ferrosilicon.

The methods of chemical analysis of various constituents of ferromolybdenum and ferrochromium are covered in a series of parts of IS 12614 and IS 13452 respectively. The chemical analysis of ferrotitanium is covered in a series of parts of this standard. With the publication of these parts, the analysis of relevant constituent prescribed for ferrotitanium in IS 1559 : 1967 will be superseded. This part covers the method for determination of carbon by the direct combustion gravimetric method. The other parts of this series are:

Part 2 Determination of silicon by gravimetric method
Part 3 Determination of titanium by the cupferron (gravimetric method)
Part 4 Determination of aluminium
Part 5 Determination of sulphur

During this revision the method for determination of carbon by the direct combustion gravimetric method has been updated.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF FERROTITANIUM

PART 1 DETERMINATION OF CARBON BY THE DIRECT COMBUSTION GRAVIMETRIC METHOD

1 SCOPE

This standard (Part 1) describes the method for determination of carbon up to 8 percent in ferrotitanium by the direct combustion gravimetric method.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
266 : 1977	Sulphuric acid (*second revision*)
1070 : 1992	Laboratory grade water (*third revision*)
1472 : 1977	Methods of sampling of ferroalloys for determination of chemical composition (*first revision*)
6226 (Part 1) : 1971	Recommendation of apparatus for chemical analysis of metals: Part 1 Determination of carbon

3 SAMPLING

3.1 The sample shall be drawn and prepared as per the method prescribed in IS 1472 : 1977.

3.2 The sample is to be cleaned with analytical grade ether and acetone, dried in an air oven at 100 ± 5°C.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed for the test.

5 DETERMINATION OF CARBON

5.1 Outline of the Method

The sample is burnt in a stream of purified oxygen and the carbon dioxide formed is absorbed, after purification, in suitable absorbent and determined gravimetrically.

6 REAGENTS

6.1 Oxygen, purity more than 99·5 percent.

6.2 Ascarite or Soda Lime, size 0·80 to 2·00 mm, dia 20 to 30 mesh.

6.3 Magnesium Perchlorate $Mg(ClO_4)_2$, anhydrous.

6.4 Boat/Crucible

Boat/crucible of precise dimensions for accommodating in the resistance/induction furnace.

6.4.1 Preignite the boats/crucibles in air or oxygen in a furnace for an hour at 1 100°C and store in a desiccator and check for consistency of the blank value.

6.5 Flux/Accelerator

Copper, red lead (preignited at 550°C), tin and iron of low carbon content.

7 APPARATUS

7.1 The apparatus recommended in IS 6226 (Part 1) : 1971 may be used.

7.2 Instead of the resistance furnace an induction furnace may also be used.

8 PROCEDURE

8.1 Assemble the apparatus. Switch on the furnace, if it is a resistance furnace, and allow it to attain a temperature of 1 250° to 1 350°C, all the while passing oxygen through the apparatus so that it bubbles freely at the exist end of the train.

Disconnect the absorption bulb, keep in a desiccator till it attains room temperature and take the initial weight. Repeat the operation till a constant mass is obtained.

8.2 Weigh to the nearest 0·001 g, 2·0 to 10·0 g for carbon content up to 3 percent and 0·25 g for carbon content above 3 percent of the test sample. Transfer to the preignited combustion boat covered at the bottom with a thin layer of calcined alumina. Spread the sample evenly over the top of the alumina and cover it with 2·0 to 8·0 g of the flux. Introduce the boat slowly in the hot zone of the combustion tube.

8.3 In the case of induction heating, weigh to the nearest 0·001 g, 0·9 to 1·1 g of the sample and transfer to a preignited crucible. Add an equal quantity of the flux. Place to crucible in position on the pedestal post of the furnace, raise to the combustion position and lock the system. Pass oxygen through the system and ignite the sample.

8.4 Maintain a rapid flow of oxygen (800 to 1 000 ml/min) throughout the combustion, then reduce to 400 to 500 ml/min and maintain it for another 6 to 8 minutes in order to sweep out the carbon dioxide.

8.5 Remove the absorption bulb and weigh it after keeping it in desiccator till it attains room temperature. The increase in mass of the bulb represents the mass of carbon dioxide.

8.6 Remove the boat or crucible and examine for any incomplete combustion. If the sample is not thoroughly fused, repeat the determination with a fresh sample.

8.7 Blank

Make a blank determination following the procedure specified in **8.2** to **8.6** and using same quantity of reagents except to sample.

9 CALCULATION

$$\text{Carbon, percent by mass} = \frac{A - B}{C} \times 27.29$$

where

A = increase of mass in g, of the absorption bulb from the sample;

B = increase of mass in g, of the absorption bulb from the blank determination; and

C = mass in g, of the sample taken.

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